

JAPANESE [JP,08-176578,A]

CLAIMS DETAILED DESCRIPTION TECHNICAL FIELD PRIOR ART EFFECT OF
THE INVENTION TECHNICAL PROBLEM MEANS OPERATION EXAMPLE

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001] [Industrial Application] This invention relates to the grease composition for anti-friction-bearing enclosure which carries out the lubrication of the sealed rolling bearing for an electric equipment article and other auxiliary machinery, such as an idler belt pulley for cars, and a tension pulley.

[0002] [Description of the Prior Art] In recent years, although the miniaturization of the electric equipment article and accessory part and the weight saving are attained with the demand of the miniaturization of a car, and a weight saving, on the other hand, the demand of high power and efficient-izing grows for the performance of a device itself, and the technique with which it compensates by carrying out the high velocity revolution of the fall of the output produced with the demand of a miniaturization is taken.

[0003] In order to prevent specifically miniaturizing the belt pulley which transmits driving force to the axis of rotation as much as possible, and decline in a transmission efficiency, many engagement grooves of a conduction belt are ganged to a belt pulley, and also means, such as making the tension of a belt high, are adopted. For the reason, the high velocity revolution of the bearing which supports a belt pulley will be carried out, and high load will be added.

[0004] In the above-mentioned grease prelubricated bearing, the life of the grease by degradation of lubrication performance is shorter than the life by fatigue of the bearing itself, and The sake, The conventional grease currently used for such an electric equipment article and the bearing for auxiliary machinery, A high temperature durability good thing is mainly chosen, and the presentation as base oil Mineral oil, Lithium soap, a lithium complex, poly urea, and a fluoro-resin were used as a thickener using synthetic hydrocarbon, a polyol-ester oil, an alkyl diphenyl ether oil, silicone oil, and a fluoride oil.

[0005] [Problem(s) to be Solved by the Invention] However, when the existing high temperature durability above-mentioned grease is enclosed with sealed bearing, an allophone

(henceforth an allophone at the time of the cold) occurs at the time of the start up under the low temperature in a cold district, and it has the problem that silence cannot be maintained.

[0006]In order for the moisture of muddy water etc. to permeate easily into the bearing for cars, it is necessary to take the rust prevention inside a bearing into consideration.

[0007]In JP,5-140576,A which the applicant of this application requires for earlier application, Although the grease composition for bearing enclosure which blended 5 to 40 % of the weight indicated the alicycle fellows diurea compound as a thickener to the base oil which blended the poly alpha olefin oil and the alkyl diphenyl ether oil by the weight ratio of 0.5 to 0.9:0.1-0.5, Room to be further improved in respect of high temperature durability was one of this thing.

[0008]Then, this invention has the character which was extremely excellent in high temperature durability (long lasting nature), moreover, suppresses generating of an allophone at the time of the cold, covers a temperature requirement wide from an elevated temperature to low temperature, and maintains the silence of sealed bearing, and it makes it the technical problem to consider it as the grease also in consideration of the rust prevention of the bearing.

[0009]

[Means for Solving the Problem]In this invention in order to solve the above-mentioned technical problem, As a thickener, blend 5 to 40 % of the weight with base oil which blended a poly alpha olefin oil and an alkyl diphenyl ether oil by a weight ratio of 0.2 to 0.8:0.2-0.8, and an alicycle fellows diurea compound. As an extreme pressure agent, a dithiophosphate salt was used as an essential ingredient and it was considered as a grease composition for anti-friction-bearing enclosure which added further a thing of dithiocarbamate or phosphoric ester which was independent respectively or carried out both mixing.

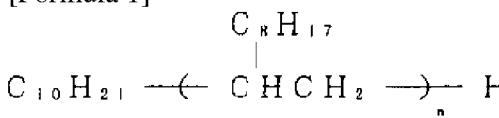
[0010]Or as a thickener, blend 5 to 40 % of the weight with base oil which blended a poly alpha olefin oil and an alkyl diphenyl ether oil by a weight ratio of 0.2 to 0.8:0.2-0.8, and an alicycle fellows diurea compound. It was considered as a grease composition for anti-friction-bearing enclosure which added two or more sorts of dithiophosphate salts chosen from dithiophosphate zinc, dithiophosphate molybdenum, and dithiophosphate lead as an extreme pressure agent.

[0011]It was considered as a grease composition for anti-friction-bearing enclosure which added a rust-proofer in the above-mentioned grease for anti-friction-bearing enclosure.

[0012]A poly alpha olefin oil used for this invention low-polymerizes an alpha olefin, is the structure which added hydrogen to that end double bond, and can illustrate what is shown in a formula of following ** 1.

[0013]

[Formula 1]



(式中、n は 1 ~ 6)

[0014]Although the alkyl diphenyl ether oil used for this invention is obtained by the addition reaction of 1-3 mol of alpha olefins of 1 mol of diphenyl ether, and the carbon numbers 10-22, that description changes with the carbon numbers and the numbers of use mols of an alpha olefin.

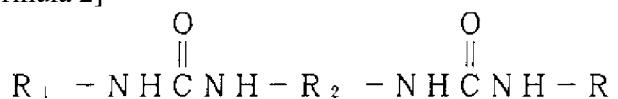
[0015]The compounding weight ratio of the poly alpha olefin oil and alkyl diphenyl ether oil in the base oil of this invention is 0.2 to 0.8:0.2-0.8. Because, with such a range, base oil viscosity (for example, kinetic viscosity:cSt in 40 **) shows a small value called about 58 cSt(s) order (55 - 60cSt), and is preferred, It is because heat resistance is fully obtained no longer and is not preferred, if the performance under low temperature which carried out expected comes to be inferior in a poly alpha olefin oil and it crosses a

mentioned range in less than a mentioned range.

[0016]Below an alicycle follows diurea compound used as a thickener in this invention is shown by formula of following ** 2, and this thing is obtained by the reaction of diisocyanate and monoamine.

[0017]

[Formula 2]



(式中、 R_2 は炭素数 7 ~ 13 の芳香族炭化水素基、 R_1 及び R_3 はシクロヘキシル基または炭素数 8 ~ 20 のアルキル基を示し、 R_1 または R_3 の少なくとも一方がシクロヘキシル基)

[0018]As diisocyanate which forms R_1 in the formula of ** 2, 4 and 4'-diphenylmethane diisocyanate, tolylene diisocyanate, etc. are mentioned. As monoamine which forms R_2 and R_3 , cyclohexylamine, stearylamine, Sept Iles amine, lauryl amine, etc. are mentioned among the formula of ** 2.

[0019]The addition to said base oil of such an alicycle follows diurea compound is 5 to 40 % of the weight. because, the viscous large quantity with scarce grease which added and obtained less than 5% of the weight of diurea compounds [a small amount of] which becomes liquefied and exceeds 40 % of the weight -- if -- it is because it becomes a solid state and becomes unsuitable as description of the grease for bearing enclosure.

[0020]In order to make base oil viscosity small as much as possible as mentioned above and to improve the grease life at the time of high temperature durability, i.e., a heatproof, in consideration of the lubrication performance of the grease in a low temperature service, it is compensating with this invention by usually adopting the predetermined extreme pressure agent which prevents oil film destruction in the friction surface of high load.

[0021]In this case, a dithiophosphate salt is used as an essential ingredient as an effective predetermined extreme pressure agent, Furthermore, dithiocarbamate, or respectively independent or two or more sorts of dithiophosphate salts which would not carry out both mixing or are chosen from dithiophosphate zinc, dithiophosphate molybdenum, and dithiophosphate lead of phosphoric ester are adopted.

[0022]As a salt of a dithiophosphate salt or dithiocarbamate, each can adopt zinc, molybdenum, or lead. . Each specifically had an alkyl group or an allyl group as a chain. Dithiophosphate zinc (zinc dithiophosphate and the following write it as Zn-DTP.), Dithiophosphate molybdenum (molybdenum dithiophosphate and the following) It is written as Mo-DTP. Dithiophosphate lead (it is hereafter written as Pb-DTP.), Zinc dithiocarbamate (zinc dithiocarbamate and the following write it as Zn-DTC.) and molybdenum dithiocarbamate (molybdenum dithiocarbamate and the following write it as Mo-DTC.) can be illustrated.

[0023]Phosphoric ester which is used by this invention and which is usually used as an extreme pressure agent is tricresyl phosphate (it is hereafter written as TCP.), and triphenyl phosphate, and these are the synthetic compounds of common knowledge compounded considering toluene or benzene as a charge of a start material.

[0024]Entire volume of a grease composition blended various kinds of extreme pressure agents described above about 0.1 to 10% of the weight, and they have obtained a desirable result.

[0025]Next, as a rust-proofer used for this invention, a sulfonate and an ester compound were used together and a desirable result has been obtained. Ammonium sulfonate, barium sulfonate, etc. are mentioned as an example of a sulfonate excellent in rust-resistor nature. Succinic acid half ester is mentioned as the ester compound. As for an addition of these rust-proofers, about 0.1 to 5 % of the weight is efficient, and is usually preferred.

[0026]

[Function]The grease composition for anti-friction-bearing enclosure of this invention serves as base oil which base oil viscosity became small at by using that base oil as the mixed oil which blended the alkyl diphenyl ether oil and the poly alpha olefin oil at a predetermined rate, and was excellent in the lubricity in a low temperature service, and was excellent also about heat resistance. As a thickener added by such base oil, thermal stability, oxidation stability, Adhesion uses the alicycle fellows diurea compound superior to metallic soap, such as lithium soap and hard soap, and by having added the further predetermined extreme pressure agent, heat resistance fully improves and becomes long lasting grease at an elevated temperature.

[0027]A sulfonate and an ester compound are added to the above-mentioned constituent, and rust prevention is remarkable.

[0028]

[Example]

[Examples 1 and 2] The base oil which consists of a mixed oil of a poly alpha olefin oil and an alkyl diphenyl ether oil is prepared by the blending ratio shown in Table 1, 1 mol of 4 and 4'-diphenylmethane diisocyanate in the liquid melted in the moiety of the base oil (% of the weight) shown in Table 1. After adding melting and agitating 2-mol monoamine (cyclohexylamine) to a moiety of remaining base oil, continue churning and it was made to react for 30 minutes at 100-120 **, and into base oil, the alicycle fellows diurea compound was made to generate, and was made and blended.

[0029]After carrying out addition mixing of the specified quantity of the extreme pressure agent and rust-proofer which add 0.5 % of the weight, agitate phenothiazin for 10 minutes at 100-120 **, are cooled after that, and are shown in Table 1 as an antioxidant, respectively, in this grease, it uniformed with 3 rolls, and grease was obtained in it.

[0030]Measurement of an allophone and a rust tightness examination were done at the time of the workability of this grease, the dropping point, an elevated-temperature durability test, and the cold, and that result was written together to Table 1.

[0031]** ** Degree: JIS K2220 It measured by 5.3.

[0032]** Drop Point: It is the temperature (**) which grease dissolves and begins to fall by self-weight Grease dropping point test-method JIS K2220 It measured by 5.4.

[0033]Elevated-temperature durability test : Enclose grease with the bearing 6204 and iron noncontact seals to both side surfaces ** Equipment, Equip a bearing driving device with this, keep ambient temperature at 150 **, and The radial road 67N, The inner ring of spiral wound gasket was rotated at the rate of per minute 10000 rotations of the axial load 67N under a condition, when the grease in a bearing deteriorated, the running torque of the bearing became excessive and time until the input current of an electric motor which is driving rotation of this principal axis exceeds limiting current and an electric motor stops was made into the life.

[0034]** the time of the cold -- allophone measurement: -- enclosing grease with the bearing 6203 -- the contact seal made of rubber -- both side surfaces -- equipment. The bearing was set to housing, after putting it into the -40 ** cryostat and making it cool enough, it attached to the bearing device installed in the room temperature, the inner ring of spiral wound gasket was rotated at the rate of per minute 2000 rotations under the conditions of the axial load 1.96N, and the existence of generating of the abnormal noise (at the time of the cold allophone) by an acoustic sense was investigated.

[0035]** A rustproof examination : the method which made still severer the rust examining method of ASTM D 1743 was adopted. That is, 1.9-2.1g of sample grease was enclosed with the tapered roller bearing 30204, axial load was added to it 98N, and the running in was carried out to it for 1 minute by per minute 1800 revolutions. Next, it was immersed for 10 seconds into a 1% salt solution. The rusting situation was investigated, after putting this bearing into the desiccator and neglecting it at 40 ** for 48 hours. The rusting situation divided the outer-ring-of-spiral-wound-gasket race into 32 equally in the hoop direction, counted the section which had rust among these, and

made n= 4 averages rust marks.

[0036]

[Table 1]

材料 または試験項目	番号			
	実	施	例	
1	2	3		
基油組成重量比	ポリαオレフィン油 (重量比)	0.2	0.2	0.2
	アルキルジフェニル エーテル油 (重量比)	0.8	0.8	0.8
	40°Cの動粘度 (cSt)	58.1	58.1	58.1
配合割合(重量%)	基油	87.5	87.5	87.5
	増稠剤(脂環族ジケ)	10	10	15
	酸化防止剤(フェノキサン)	0.5	0.5	0.5
	極圧剤 Mo-DTP	0.5	0.5	0.5
	Zn-DTP	—	0.5	—
	Mo-DTC	—	—	0.5
	リチ酸エチル(TCP)	0.5	—	—
	防錆剤 アモニウムホメート	0.5	0.5	0.5
	コハク酸ハーフエチル	0.5	0.5	0.5
稠度		290	290	288
滴点(°C)		255	252	258
高温耐久試験 寿命時間;h		>4000	>4000	>4000
冷時異音発生の有無		無	無	無
錆防止試験(n=4)で錆評点		2	0	1

[0037] [Comparative examples 1-3] As shown in Table 2, said predetermined blending ratio was exceeded, what mixed alkyl diphenyl ether (10 % of the weight) with the poly alpha olefin oil (90 % of the weight) was used as base oil, and grease which used alicycle fellows diurea for the thickener was made into the comparative example 1. What mixed the Polly alpha olefin oil (80 % of the weight) and the alkyl diphenyl ether oil (20 % of the weight) was used as base oil, and grease which adopted aromatic diurea as the thickener was made into the comparative example 2. Aromatic diurea was adopted as the thickener, the completely same examination ** as an example - ** were performed by having made into the comparative example 3 grease which used base oil as the independent ingredient of an alkyl diphenyl ether oil, and the result was written together all over Table 2.

[0038]

[Table 2]

材料 または試験項目	番号	比 較 例		
		1	2	3
基油 組成 重量 比	ポリ α オレフィン油 (重量比)	0.9	0.2	0
	アルキルジフェニル エーテル油 (重量比)	0.1	0.8	1
	40°Cの動粘度 (cSt)	52.5	72.3	123.0
配合 割合 (重量 %)	基 油	77.5	74.5	74.5
	增 稠 剤	20	—	—
	芳香族ジウレア	—	23	23
	酸化防止剤 (ワクアツジ)	0.5	0.5	0.5
	Mo-DTP	0.5	0.5	0.5
	TCP	0.5	0.5	0.5
	防 錆 剤	アモニウムホネート	0.5	0.5
	ヨウ酸マグネシウム	0.5	0.5	0.5
稠 度	275	300	260	
滴 点 (°C)	>260	240	>260	
高温耐久試験 寿命時間:h	560	2100	>4000	
冷時異音発生の有無	無	有	有	
錆防止試験(n=4)の錆評点	0	1	1	

[0039][Comparative examples 4-6] As shown in Table 3, in the grease which added the same antioxidant using the completely same base oil as the actual example 1, and a thickener. Grease which added only any one 1% of the weight was made into the comparative examples 4-6 out of dithiophosphate molybdenum (Mo-DTP), molybdenum dithiocarbamate (Mo-DTC), and tricresyl phosphate (TCP).

[0040] To these, the completely same examination ** as an example - ** were performed, and the result was written together all over Table 3.

[0041]

[Table 3]

材料 または試験項目	番 号			
	4	5	6	
基油組成重量比	ポリαオレフィン油 (重量比)	0.2	0.2	0.2
	アルキルジフェニル エーテル油 (重量比)	0.8	0.8	0.8
	40°Cの動粘度 (cSt)	58.1	58.1	58.1
配合割合 (重量%)	基 油	87.5	87.5	87.5
	増稠剤 (脂環族オレフ)	10	10	10
	酸化防止剤 (フェノキサン)	0.5	0.5	0.5
	極 壓 剤 Mo-DTP	1.0	—	—
	Mo-DTC	—	1.0	—
	リン酸乙チル (TCP)	—	—	1.0
	防 鏽 剤 アモニウムスルホネート	0.5	0.5	0.5
	コク酸ヘプチカル	0.5	0.5	0.5
稠 度	282	283	290	
滴 点 (°C)	255	256	253	
高温耐久試験 寿命時間:h	1560	1780	1350	
冷時異音発生の有無	無	無	無	
錆防止試験(n=4)で錆評点	1	1	1	

[0042][Comparative examples 7-9] As shown in Table 4, grease which added either ammonium sulfonate, barium sulfonate or succinic acid half ester 1% of the weight in the grease which added the same extreme pressure agent as the same antioxidant, respectively was made into the comparative examples 7-9 using the completely same base oil as the actual example 1, and a thickener.

[0043]To these, the completely same examination ** as an example - ** were performed, and the result was written together all over Table 4.

[0044]

[Table 4]

材料 または試験項目	番号			
	7	8	9	
基油組成重量比	ポリαオレフィン油 (重量比)	0.2	0.2	0.2
	アルキルジフェニル エーテル油 (重量比)	0.8	0.8	0.8
	40°Cでの動粘度 (cSt)	58.1	58.1	58.1
配合割合 (重量%)	基油	87.5	87.5	87.5
	増稠剤 (脂環族アリケ)	10	10	10
	酸化防止剤 (フェノチアゾン)	0.5	0.5	0.5
	極圧剤 : Mo-DTP	0.5	0.5	0.5
	ツル酸エチル (TCP)	0.5	0.5	0.5
	防錆剤	アモニウムズホネート	1.0	—
		バウムズホネート	—	—
		コハク酸ハーフエチル	—	1.0
	潤度	278	276	276
	滴点 (°C)	256	258	256
高温耐久試験 寿命時間:h		960	1120	480
冷時異音発生の有無		無	無	無
錆防止試験(n=4)での錆評点		18	28	2

[0045]The comparative example 1 and the comparative example 3 besides said predetermined range were inferior to high temperature durability in the blending ratio of a poly alpha olefin oil and an alkyl diphenyl ether oil, or the allophone occurred at the time of the cold so that clearly from the test result of Table 2. The comparative example 2 uses aromatic diurea for the thickener, although a base oil presentation is predetermined within the limits.

The allophone occurred at the time of the cold.

[0046]High temperature durability all has not fully improved the comparative examples 4-6 which blended only any one sort of DTP, DTC, or phosphoric ester as an extreme pressure agent so that clearly from the result of Table 3.

[0047]The comparative examples 7-9 which adopted only any one sort of a sulfonate salt or the ester compound as a rust-proofer were inferior to rust prevention, or even if they satisfied rust prevention, they were inferior to high temperature durability, so that clearly from the result of Table 4.

[0048]On the other hand, Examples 1-3 with which it is further satisfied of the predetermined addition conditions of an extreme pressure agent by making a base oil presentation into a predetermined blending ratio, using an alicycle fellows diurea

compound as a thickener so that clearly also from the result of Table 1, All showed the outstanding character in which high temperature durability was 4000 hours or more, and did not have generating of an allophone further at the time of the cold, either.

[0049]These examples do a remarkable rust prevention effect so, without reducing said outstanding high temperature durability by using a sulfonate and an ester compound together.

[0050]

[Effect]In this invention, as explained above, the mixed base oil which blended the alkyl diphenyl ether oil and the poly alpha olefin oil at a predetermined rate as base oil, and made viscosity small as much as possible is adopted, And the alicycle follows diurea compound excellent in heat resistance was used as a thickener, and it was considered as the grease for anti-friction-bearing enclosure which adopted the further predetermined extreme pressure agent.

Therefore, it has the character which was extremely excellent in high temperature durability (long lasting nature), moreover, generating of an allophone is suppressed at the time of the cold, and there is an advantage which can cover a temperature requirement wide from an elevated temperature to low temperature, can maintain the silence of sealed bearing, and serves as grease.

[0051]It can be said that what added the rust-proofer in such grease can contribute to reinforcement of anti-friction bearing which enclosed such grease greatly, and its industrial utility value is very high since rust prevention also appears notably in addition to the above-mentioned advantage.

[Translation done.]